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LEED-AES-THIN LAYER ELECTROCHEMICAL STUDIES OF HYDROGEN ADSORPT--ETC(U)  
AUG 82 A S HOMA, E YEAGER, B D CAHAN

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LEED-AES-THIN LAYER ELECTROCHEMICAL STUDIES OF HYDROGEN  
ADSORPTION ON PLATINUM SINGLE CRYSTALS

by

Arthur S. Homa, E. Yeager and B.D. Cahan

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Case Center for Electrochemical Sciences  
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Case Western Reserve University  
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20. Abstract, continued

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ABSTRACT

The understanding of the relation of electrocatalysis to electrode surface structure requires measurements on electrode surfaces free of impurities and of well defined morphology. A system has been developed which allows preparation and characterization of such surfaces in an ultrahigh vacuum using LEED and AES, their electrochemical examination within a thin-layer cell, and subsequent re-examination of the surface morphology and purity. The electrochemical adsorption-desorption of hydrogen has been studied on the three low index planes of Pt in dilute HF. When the potential range is restricted to 0.05-0.5 V vs RHE (1 atm), post-electrochemical LEED analysis shows that the Pt(111) and (110)-(1x2) surfaces retain their original structure while the Pt (100)-(5x20) loses its 5x20 overlayer.

I. Introduction

In an effort to establish the effect of surface morphology on the electroadsorption of hydrogen, various workers have examined the adsorption-desorption of hydrogen on single crystal platinum surfaces (1-12). A severe problem in such studies is the achieving of clean platinum surfaces of known surface topography in electrolytes. Well characterized single crystal surfaces prepared in ultra high vacuum are easily contaminated during the transfer into the electrochemical environment and can undergo restructuring particularly if cycled to relatively anodic potentials where anodic film formation occurs. Most workers using platinum single crystals in such studies have usually cycled their electrodes to relatively anodic

potentials on the basis that such procedures are necessary to remove impurities before examining the hydrogen voltammetry. Without such cycling, the hydrogen region of the voltammetry curves usually does not yield well defined peaks in either polycrystalline or single crystal platinum.

## II. Experimental Procedures and Apparatus

To overcome these problems, the present study has made use of the specially designed LEED-Auger ultrahigh vacuum system shown in Fig. 1, which is coupled with a thin-layer electrochemical cell system. Well ordered single crystal surfaces are prepared and cleaned by standard surface physics techniques and characterized by LEED and AES in UHV ( $5 \times 10^{-11}$  Torr base pressure) in the right-hand chamber. The electrodes are then transferred to a second UHV chamber B by a magnetically coupled manipulator without loss of UHV integrity. A thin-layer cell is then formed in chamber B using an  $\alpha$ -Pd/H counter/reference electrode prepared in a third UHV chamber attached to the system. Introduction of ultra-pure argon into the chamber B provides an atmosphere for wet electrochemical studies. An all-Teflon electrolyte delivery system allows dilute HF (e.g., 0.1 M) to be used as the electrolyte. HF offers the advantage that the electrolyte components are not specifically adsorbed and the electrolyte is completely volatilizable. This allows for post-electrochemical analysis of the surfaces by LEED and AES without a contaminating surface overlayer.

The electrochemical cell (Fig. 2) employed in these studies is a two-electrode thin-layer design which minimizes contamination problems in the electrolyte with a large electrode area-to-solution volume ratio. This design also allows for ease of manipulation. The electrolyte is first applied to the horizontal surface of the  $\alpha$ -Pd/H counter/reference electrode as a single drop and then raised to bring the electrolyte into contact with the

single crystal electrode under potential control in chamber B with externally coupled magnetic manipulators. Following the electrochemical examination, the two electrodes are separated and the small amount of electrolyte volatilized as the pressure is rapidly pumped down to  $\sim 10^{-8}$  Torr in chamber B with a turbomolecular pump. The electrode is then returned to chamber A where it is re-examined with LEED and AES at a pressure of  $\sim 5 \times 10^{-11}$  Torr. The total time from separation of electrodes to return to this pressure is  $\sim 5$  minutes.

The platinum single crystals used in the present study were cut from Marz grade single crystal rod (Materials Research Corp.). The desired single crystal face was aligned to within  $1^\circ$  by Laue X-ray backscattering and spark cut into a 1-mm thick slice. The 6.4-mm diameter disks were spot welded to a 1.3-cm diameter Pt foil disk which in turn was spot welded to a specifically designed molybdenum sample holder. The platinum surface was then polished using standard metallurgical techniques with the final polishing step involving  $0.05 \mu\text{m}$  alumina.

The single crystal sample was then subjected to alternate cycles of argon ion bombardment and high temperature ( $1000^\circ\text{C}$ ) annealing in vacuum chamber A at a base pressure of  $5 \times 10^{-11}$  Torr until the LEED pattern characteristic of the specific surface was obtained and the AES spectrum showed no trace of impurities, particularly C and O.

The  $\alpha\text{-Pd/H}$  counter reference electrode was charged with hydrogen electrochemically using an ancillary platinum electrode in a third chamber attached to chamber B with great care to obtain a surface free of any impurities. This electrode has a potential of  $+0.05$  V vs. the reversible hydrogen electrode ( $p_{\text{H}_2} = 1$  atm) in the same electrolyte. No introduction of Pd into the electrolyte occurs at this potential.

### III. Results

#### LEED Studies after Electrochemical Treatment

In the voltammetry studies of  $H_{ads}$  on single crystal Pt surfaces with this apparatus, the potential sweep range has been restricted to relatively cathodic potentials [e.g., 0.05 V vs. RHE ( $p_{H_2} = 1$  atm)] to reduce the possibility of restructuring of the surface. Subsequent LEED examination for the low index surfaces indicates that the essential features of the surface are preserved although there is some increment in background scattering (Fig. 2). If the voltage sweep is extended into the anodic film formation region (1.4 V), the hydrogen adsorption region is substantially changed with new peaks appearing or minor peaks becoming major, depending on the particular low index surface (see Fig. 3). Post-electrochemical LEED examination reveals loss of pattern for low beam voltage and hence loss of longer range surface ordering over the coherence length of the electron beam (30-50 nm).

The Pt(100) surface prepared by high temperature annealing has a (5x20) overlayer structure. Post-electrochemical LEED analysis (Fig. 2a,b) indicates loss of this overlayer structure after exposure of the electrode to the 0.1 M HF with potential cycling in the range 0.05-0.5 V. The Pt(110) surface initially has a 2x1 structure consisting of (111) oriented microfacets and retains this structure throughout the electrochemical measurements in the range 0.05 - 0.5 V (see Fig. 2c,d).

Cycling any of the low index surfaces to 1.4 V resulted in a major deterioration of the LEED pattern with the spot pattern becoming evident only at much higher electron beam voltages (>100 eV) compared to those (~60-80 eV) used in Fig. 2. The higher beam voltages result in more sensitivity to sub-layer. The implication is that the surface disorder caused by anodic film formation and reduction is limited to the first or second layer.

The post-electrochemical AES examination of the electrodes, cycled only in the region 0.05 - 0.5 V and also those cycled in the range 0.05 - 1.4 V, has indicated not more than 10% of a monolayer of carbon and no other contaminants (10). This small amount of carbon may explain the increment in background scattering in the post-electrochemical LEED examination. The same carbon surface contamination level has been found when the Pt was exposed only to the argon atmosphere without introduction into the electrolyte. The carbon is either present in the argon despite the purification procedures or, more likely, is released from the walls of the vacuum chamber during pumpdown.

#### Hydrogen Adsorption-Desorption

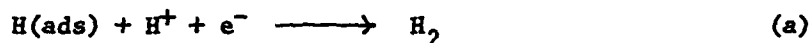
The cyclic voltammetry curves for the hydrogen region on the three low index surfaces are shown in Fig. 4. The (111) and (110) surfaces of Pt exhibit similar behavior as may be expected in view of the 2x1 overlayer structure on the (110) surface, which appears to persist even after the electrochemical environment (Fig. 2c,d). After deconvolution out of the  $H_2/H^+$  faradaic components, there is only one pronounced peak on the Pt(110) and Pt(111) surfaces, corresponding to the weakly adsorbed hydrogen. Hubbard et al. (4) and Ross (5) also found one predominant peak on these surfaces. The charge under the single peaks in Fig. 4A,B in the present work, however, is much less than found by these other workers, for example, on the (111) surface, ~35% of that expected for a monolayer, using one electron per surface Pt as corresponding to a monolayer. With cycling to anodic potentials of 1.4 V, our results on the (111) and (110) surfaces including the charge under this peak become similar to those of Hubbard et al. and Ross.

We believe this is due to a structural change in the surfaces. An alternate explanation is that our surfaces have some impurity such as carbon blocking some of the sites and that this impurity is stripped off with cycling to such positive potentials. The 10% of a monolayer of carbon picked up in the post-electrochemical AES examination does not seem sufficient to reduce the hydrogen coverage to  $1/3$ , even if the carbon is present during the electrochemical measurements rather than corresponding to CO picked up on the surface during the pumpdown following the electrochemical measurements.

The 35% coverage on the Pt(111) surface fits in well with a model in which the hydrogen is adsorbed on three-fold sites as shown in Fig. 5. Such a model for H adsorption on Pt(111) is supported by the SCF-X $\alpha$ -scattered wave method calculations of Messmer (13) for the interaction of hydrogen atoms with Pt<sub>4</sub> tetrahedral clusters with relativistic corrections. The hydrogen atoms are in the interstices of the tetrahedral Pt<sub>4</sub> cluster. Photoemission data of Demuth (14) for the Pt(111) surface provides experimental evidence supporting this model.

Hydrogen adsorption on Pt(100) is quite different than on the other two low index surfaces. Most conspicuous is the unique appearance of a sharp cathodic spike in the anodic branch of the voltammetry curve (Fig. 4A). This spike exhibits a dependence on the potential limit of the voltammetry sweep and has been observed in HF but not H<sub>2</sub>SO<sub>4</sub> as the electrolyte (1). This anomalous behavior is not easily explained by any contamination effect or peculiar cell behavior. A likely explanation is restructuring of the Pt surface accompanied by a readsorption of a strongly bound hydrogen. Voltage window opening voltammetry measurements provide evidence that this additional strongly adsorbed hydrogen can be desorbed in the cathodic sweep

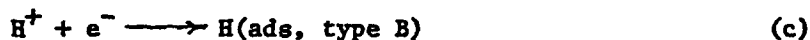
by a cathodic reaction of the form



at 0.1 V vs RHE or in the anodic sweep by anodic desorption



at the surprisingly anodic potential of 0.8 V (see Fig. 6). Reaction (a) corresponds to the ion-plus-atom reaction for  $\text{H}_2$  generation. An alternate to reaction (a) could be



followed by

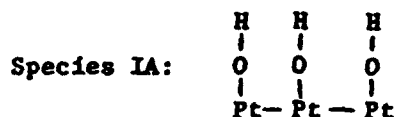


where type A adsorbed hydrogen corresponds to that electrosorbed at more cathodic potentials (~0.1 V) but then almost immediately desorbed by reaction (d).

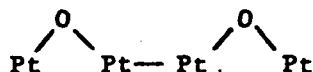
#### Effects of Anodic Cycling on Surface Structure

Despite the disordering apparent in the post-electrochemical LEED patterns, the surface structure after cycling to 1.4 V is still quite specific to the crystal orientation. Evidence for such is the specificity of the hydrogen region of the voltammetry curves to the original crystal orientation after such cycling. Just how this specificity is maintained is a matter of conjecture.

Most workers believe that Pt is covered with close to a monolayer of PtO species at 1.4 V. Horkans et al. (15) proposed the film at 1.1 V to be either



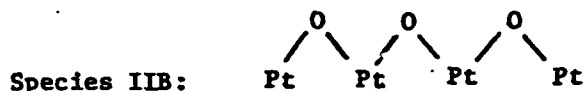
or Species IB:



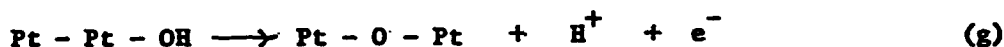
As the potential is driven more anodic, the following proposed species increase in coverage:



or



Such adsorbed layers would not necessarily be expected to lead to disordering of the Pt upon reduction. The Pt atoms must shift their positions for such disordering to occur. It is easier to envision this if some form of place exchange is involved in the anodic film formation, e.g.,



Angerstein-Kozlowska et al. (16) have proposed such place exchange. During this process, the Pt is a mobile species and this can lead to disordering.

While the LEED patterns on the low index planes show no distinguishable features for low beam energy after cycling to 1.4 V, the reduction of the anodic film may still lead to short range local ordering which depends on the crystallographic orientation. This would explain the specificity of the hydrogen adsorption-desorption region and the anodic film regions to the crystallographic orientation even after cycling to 1.4 V.

#### Acknowledgment

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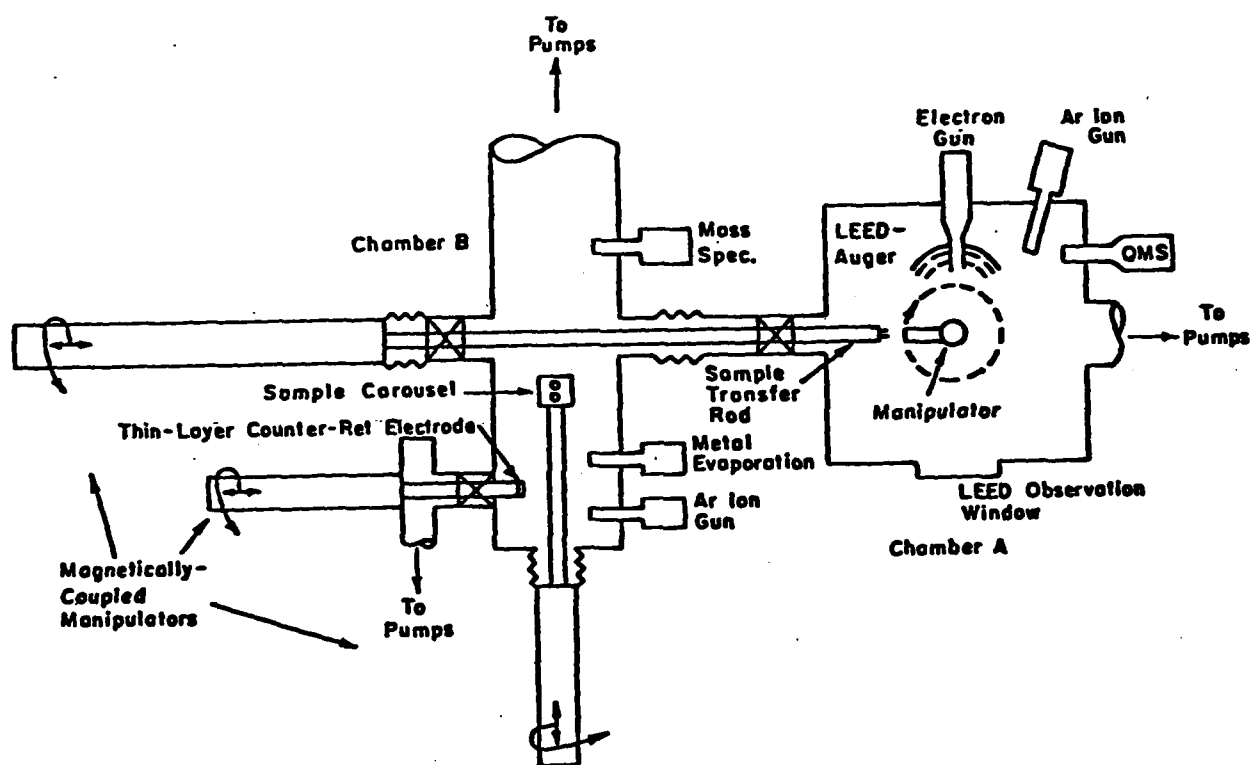


Fig. 1. LEED-Auger-thin layer electrochemical cell system with special transfer system. (17)

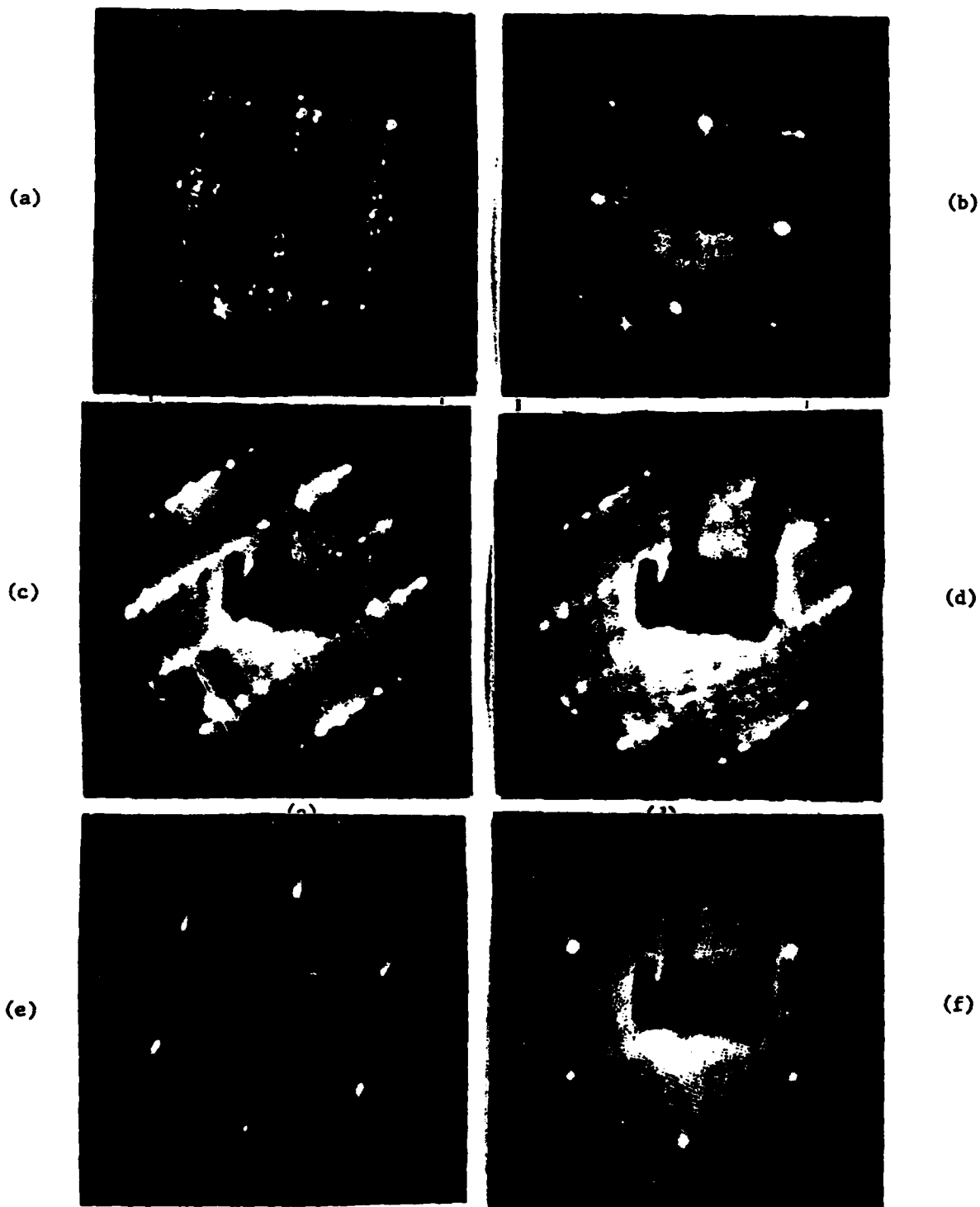


Fig. 2. LEED pattern<sup>10</sup> for Pt single crystal electrode surfaces before and after three cyclic potential sweeps from 0.5 to 0.05 V vs RHE (1 atm) in 0.1 M HF with removal at 0.05 V. Sweep rate: 50 mV/s. (a) initially Pt(100); (c) initially Pt(110); (e) initially Pt(111); (b), (d), (f) corresponding surfaces following electrochemical measurements. (17)

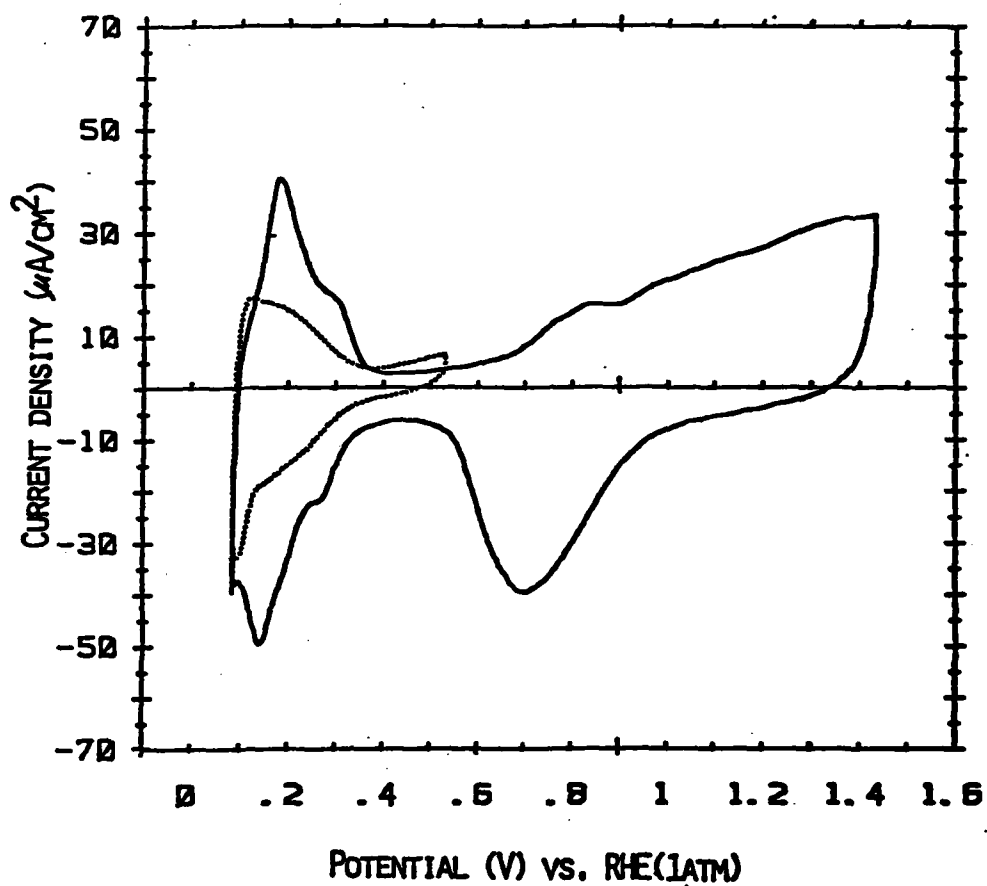


Fig. 3. Cyclic linear sweep voltammetry curve for Pt(111) in 0.1 M HF. Sweep rate: 50 mV/s. Solid line: second cycle 0.05 to 0.5 V; dotted line: fourth cycle 0.05 to 1.4 V.

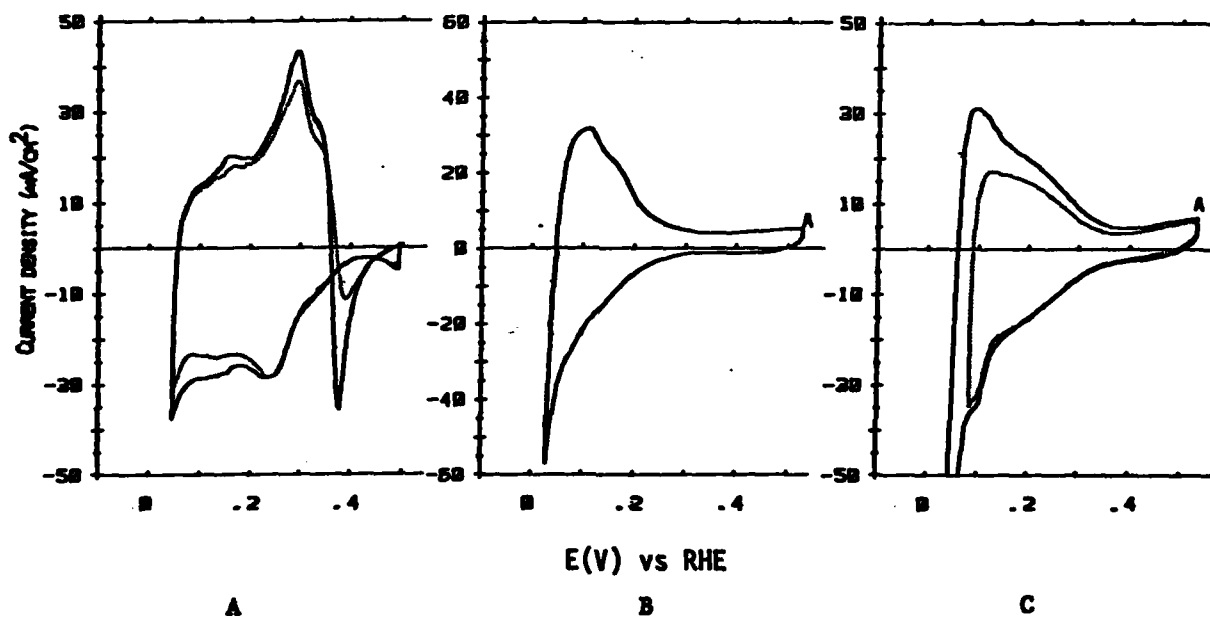
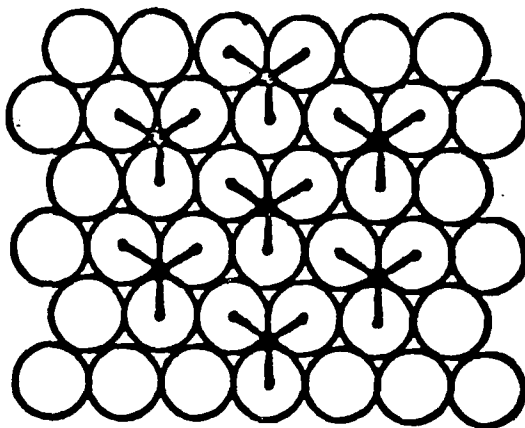


Fig. 4. Cyclic voltammetry curves in 0.1 M HF, sweep rate = 50 mV/sec.  
A. Pt(100), B. Pt(110), C. Pt(111).



**Fig. 5.** Schematic representation of three-fold sites for hydrogen adsorption on the Pt(111) surface.

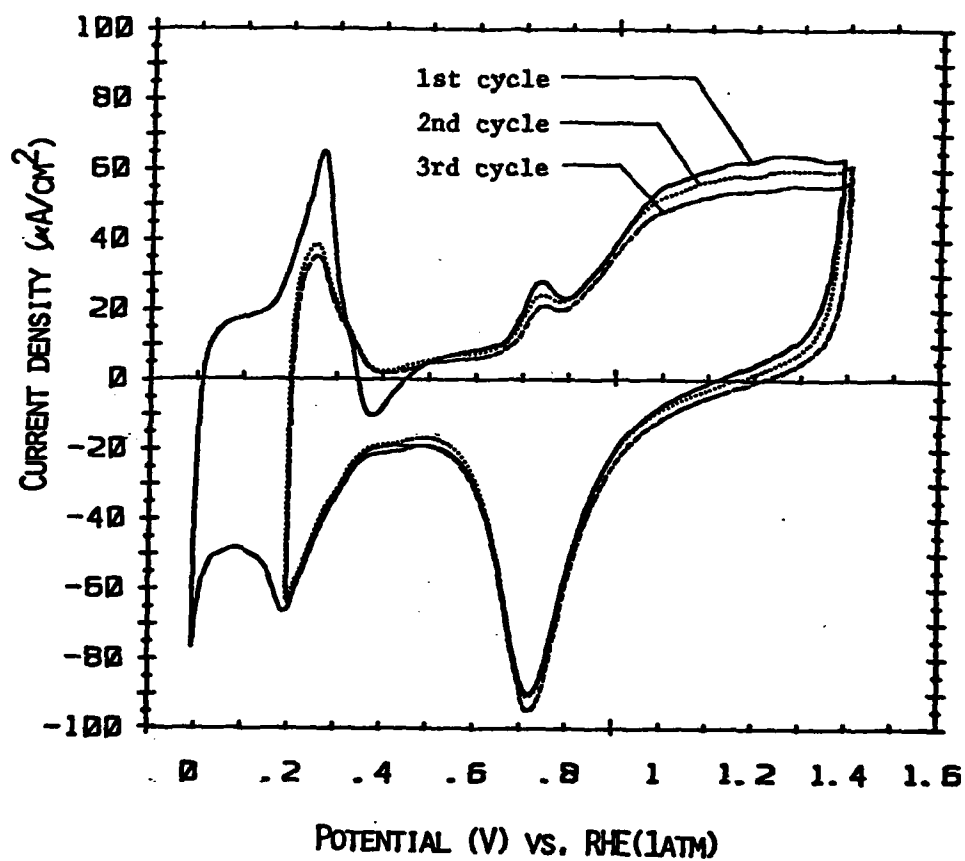


Fig. 6. Cyclic voltammetry curve for Pt(100) in 0.1 M HF.

Sweep rate = 50 mV/sec.

— First cycle 0.05 - 1.4 V.

..... Second cycle

--- Third cycle

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